AN ULTRASONIC F-SCAN INSPECTION TECHNIQUE FOR THE DETECTION OF SURFACE PREPARATION VARIANCES IN ADHESIVELY BONDED STRUCTURES

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INTRODUCTION

An adhesive bond can be used to join metal to metal, metal to composite, composite to composite, metal to honeycomb, composite to foam, etc., to form a new structure. These adhesively bonded joints are being utilized in airframes to meet the general industry requirements of increased performance and decreased weight. These bonds are replacing rivets and other mechanical fasteners in load bearing situations and failure of the bond can therefore lead to failure of the component and possibly an entire structural system.

Many variables can affect the ultimate strength of an adhesive bond (1). A brief list of variables to point out the difficulties anticipated in the development of a complete inspection procedure is presented next. Classical variables include: bondline thickness, joint type and geometry, shear and tensile modulus of adhesive, composition of adhesive with respect to the base and accelerator ratio, loading strain rate effects, surface preparation and roughness, plastic and elastic substrate qualities, humidity variation in manufacture, electrical and thermal properties of the adhesive and adherend, interfacial resistance properties, substrate surface free energy, and residual stress in the adhesive (2). Recent improvements in adhesive materials and manufacturing/quality control procedures have reduced the effects of many of these variables. The variables still of prime importance deal with the surface preparation, specifically the size of the pores in the substrate, adhesive primer thickness and the occurrence of foreign ions on the surface (3). Checking all of these individual manufacturing operations and quality control variables is almost impossible. The goal is therefore to take a measurement after the bond is completely manufactured and then to determine its overall quality.

SURFACE PREPARATION AND BONDING TECHNIQUE

In the quality/process control of adhesive joints, the determination of substrate preparation quality is most critical. Poor surface preparation usually results in a weak bond. To understand the anomalies associated with surface preparation, the basic steps in the process must be reviewed. First, large deposites of dirt and grease are manually removed from the substrate material by using an alcohol solution. This is followed by a condensing tricloro-ethylene cleaning for uniform dirt removal. An alkaline wash is used to remove contaminates and the substrate is washed in distilled water. Since aluminum oxidizes quite quickly in air, the existing oxide layer is removed. The oxidation removal solution is sulfuric acid with chromium ions (Cr^{+6} Cr^{-2}) and also small amounts of copper and aluminum. This process, called sulfachromique pickling, removes the oxide layer and introduces a 400 Angstrom layer of copper and pure aluminum onto the surface. After thoroughly rinsing the specimen, a 2 μm oxide layer is put back on the substrate using an anodizing technique. The presence (or omission) of the copper and aluminum on the surface greatly modifies the final surface condition after anodization. Figure 1 shows an electron scanning microscope image and corresponding three dimensional drawing of the resulting surfaces after anodization for the good (with Cu+Al) and the bad with out (Cu+Al) sulfachromique pickling. The multiple small pore model has slightly enhanced adhesion qualities and better resistance to environmental attack. This is the final modification of the substrate surface. A specified amount of primer is then uniformly sprayed on the surface to obtain a $3.0 \pm 1.0 \mu m$ layer. Finally, the FM73.M06 adhesive is placed between the substrate material and cured in an autoclave under vacuum conditions.

PHYSICAL MODEL

The physical model used to understand the ultrasonic signal response was a layered model with acoustic impedance variations and wave absorption at the aluminum-adhesive interface. Diagrams of the models that were used to describe the good and bad surface preparations are modified versions of the models used in (4). Three situations could occur at the interface. First, the ideal bond situation is an aluminum-adhesive interface with an extremely thin primer layer or possibly the primer diffused into the adhesive. This interfacial condition has the greatest potential for excellent strength and durability. The second situation is a small air gap or discontinuity that can occur in the pores of the substrate This is a common occurence in adhesive bonds (1) and the only way to avoid this is to prepare the specimen in a vaccum which is impractical. The third situation is an aluminum-primer-adhesive layer. This can occur if the primer collects in the larger pores of the poorly prepared surface specimen.

Theoretical analysis can be used to estimate the possible ultrasonic response function for various frequency input waves as they interact with these three interfacial models. One method to estimate the possible ultrasonic response function is to calculate the reflection factor variation as a function of frequency for these three situations using Brekhovskikh's layered media solution. Wave cancellation effects in the thin layers can cause changes in overall reflection factors for different frequency input waves. The first situation, the potentially good aluminum adhesive interface, has a constant reflection factor value of .78 for all frequencies. For the air gap model, all energy is reflected for even a .1 μm gap except for frequencies below 40 Hz. To an ultrasonic wave, the aluminum-primer-epoxy layer responds like a low pass filter with the higher frequency components being attenuated or

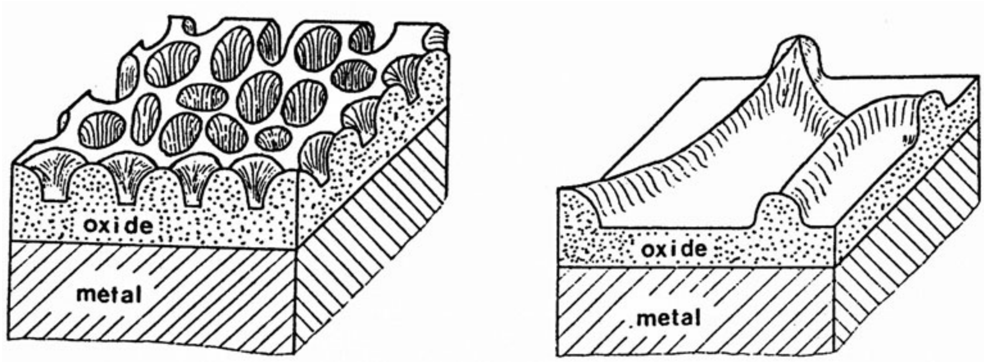

Figure 1: A model of the surface condition associated with proper (left) and inproper (right) acid etching and anodizing.

cancelled out (see Figure 2). The exact frequency range that this pass filter effects is difficult to estimate because of the simplifications made in the analysis and the physical model. Other physical models and analysis techniques could be considered, however this approach is considered sufficient with feedback from experimental results from real specimens to specify the actual frequency range.

EXPERIMENTAL

The goal of the experiment was to detect the change in interfacial quality that can result from variations in the surface preparation and environmental degradation. A series of test specimens were developed based on the geometry of the inservice structure. The specimens were 200 mm x 25 mm. The aluminum was type 2024. The adhesive was FM73-M06, which is an epoxy adhesive with chopped fibers. The nominal bond thickness is an actual structure .3mm± .1mm. However, the preparation of these specimens yielded bond layers less than the minimum .2mm at the ends. Therefore, only the thicker area was selected for nondestructive evaluation which were the middle 200mm.

Experiments were carried out in two steps. The first experiment was a feasibility study comparing a good surface preparation specimen to a poor specimen. After experimentation, the specimens were destructively tested for further information. To verify the results, one specimen with good surface preparation and five specimens with poor surface preparation were prepared. These were evaluated using a similar ultrasonic test setup.

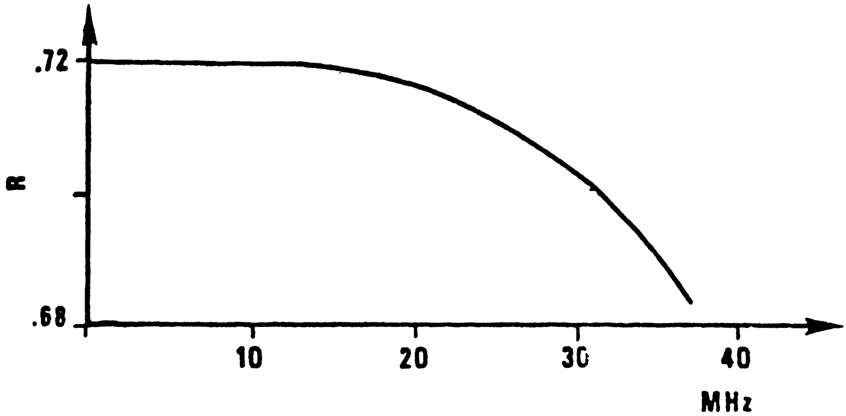

Figure 2. Brekhovskihk Lyered Media result for the poor surface preparation showing increased wave cancellation above 20 MHz.

A longitudinal ultrasonic wave was used to inspect the bonded specimen. The application of this bond dictated that only pulse echo testing could be utilized since only the nonbonded surface of the 1.6mm panel was accessible for inspection. The longitudinal wave speed in the aluminum adherend was .635 cm/μsec. The wave speed of the adhesive layer is variable since it is a function of cohesive bond strength, however, a measured wave velocity of .25 cm/μsec was used to estimate arrival times. The time between the front wall echo (FWE) and the first bond line echoes (BLE1) was .50 microseconds (μsec). Because of the variable bond thickness (.2 -.4mm) and wave speed, the arrival time of the second bond line echo (BLE2) could vary between .66 to .82 μsec. The time in the thin substrate (.6mm) is .19 μsec, so the arrival time of the backwall echo (BWE) could occur between .85 to 1.01 μsec. The second internal echo in the first substrate would occur a 1.01 μsec, the same time as the BWE, which make analysis to either of these reflections very difficult since two waves are superimposed. Also, in the application of the joint, a sealer is applied to the back surface that will modify the reflection characteristics of the interface.

A new ultrasonic specification was developed utilizing short wavelengths to try to detect the change in surface preparation. The most important component was a special higher frequency transducer with a center frequency of 30 MHz and -12dB bandwidth limits from 20 MHz to 40 MHz. This was highly focused so that the beam width was .001 in. (.025mm). Figure 3 shows a schematic of the transducer. To obtain a piezoelectric crystal to the proper thickness, a thicker crystal is first bonded to a buffer rod. Then precise grinding and polishing is performed to obtain the proper thickness. The focussing lens is attached to the buffer rod and damping material applied to the back of the crystal. The result is a high frequency focused probe that has a limited inspection window because of the interference of internal echoes in the buffer rod and lens. The return echoes were digitized at 500 MHz using a Tektronix 7912 digitizing unit. This unit can record 512 data points, or 1.02 μsec of data, which means some of the BWE will be eliminated for thicker bonds, but this was not a problem since only FWE, BLE1 and BLE2 was required for evaluation purposes. The transducer was

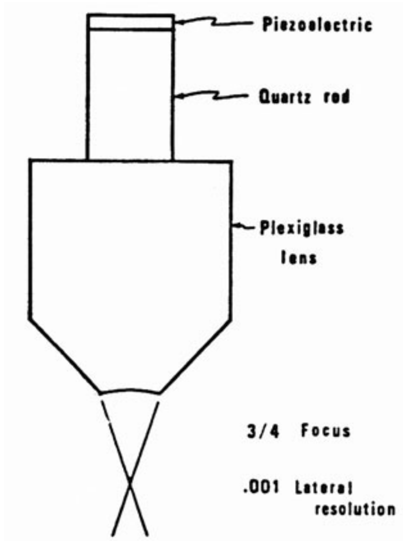

Figure 3: Diagram of the 30 MHz highly focussed transducer.

positioned over specific locations on the specimens in a grid pattern with a spacing of 2.5mm.

RESULTS ON SURFACE PREPARATION QUALITY EVALUATION

The first experimental result in the inspection of adhesively bonded joints was the successful identification of areas with good and bad surface preparation differences between the two surface preparation types were observed in the frequency area ratio features. The feature used was:

$$\text{FAR} = \frac{\text{AREA [30.0MHz - 38.0MHz]}}{\text{AREA [22.0MHz - 30.0MHz]}} \qquad [1]$$

The probability density function curve, Figure 4, shows a shift in feature values for the bad surface preparation specimen. The F-map, Figure 5, shows an area where the bad surface perparation occured. The peel test of this specimen revealed no significant difference in strength as compared to the good bond. These results correlate well with the expectations derived from the physical model.

The conclusion drawn from these results was that the small pores of the well prepared specimens were too small to absorb or scatter the high frequency waves. However, the large pores of the poor surface preparation specimen were detectable with frequencies greater than 30 MHz.

The second stage of the experiment was to verify these results. The data demonstrated the same general trend; however poor surface preparation specimens did not demonstrate the same uniformity. The probability density function curves for three selected poor surface preparation of specimens (M8A, M8B, M8C) versus the good surface preparation specimen (B2A) show large spread for the poor surface preparation specimens which is slightly exaggerated because of the convergence difficulties when trying to estimate a probability density function on a data set that is fairly random, and has a limited number of data points. The feature maps in Fig. 6 further exemplify this point. The conclusion drawn from these experiments is that lower and random values in this feature point to poor surface preparation.

CONCLUSIONS

The F-map process has proven useful in the inspection of adhesively bonded joints. The detection of surface preparation error with the 30 MHz focused transducer demonstrates the potential utility and flexibility of the F-map system. Additional testing must be carried out however, to confirm our final conclusions on detection of surface preparation errors.

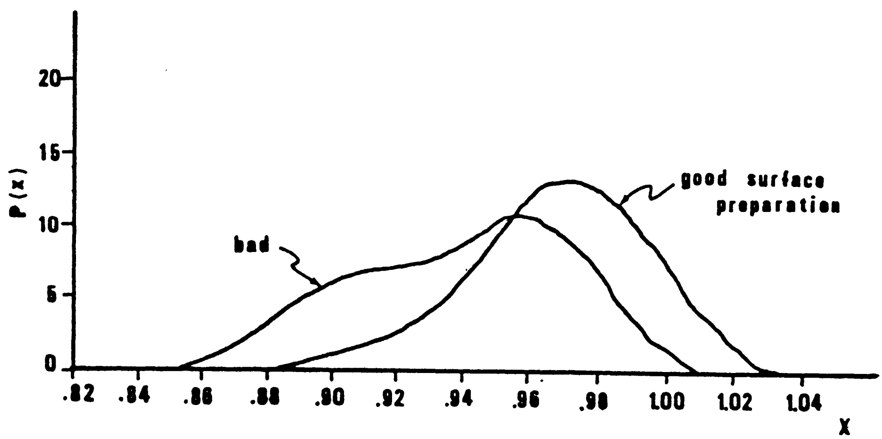

Figure 4: Probability Density Function Curve of Frequency Area Ratio. (FAR [30.0 38.0 MHz]/[22.0 30.0 MHz].

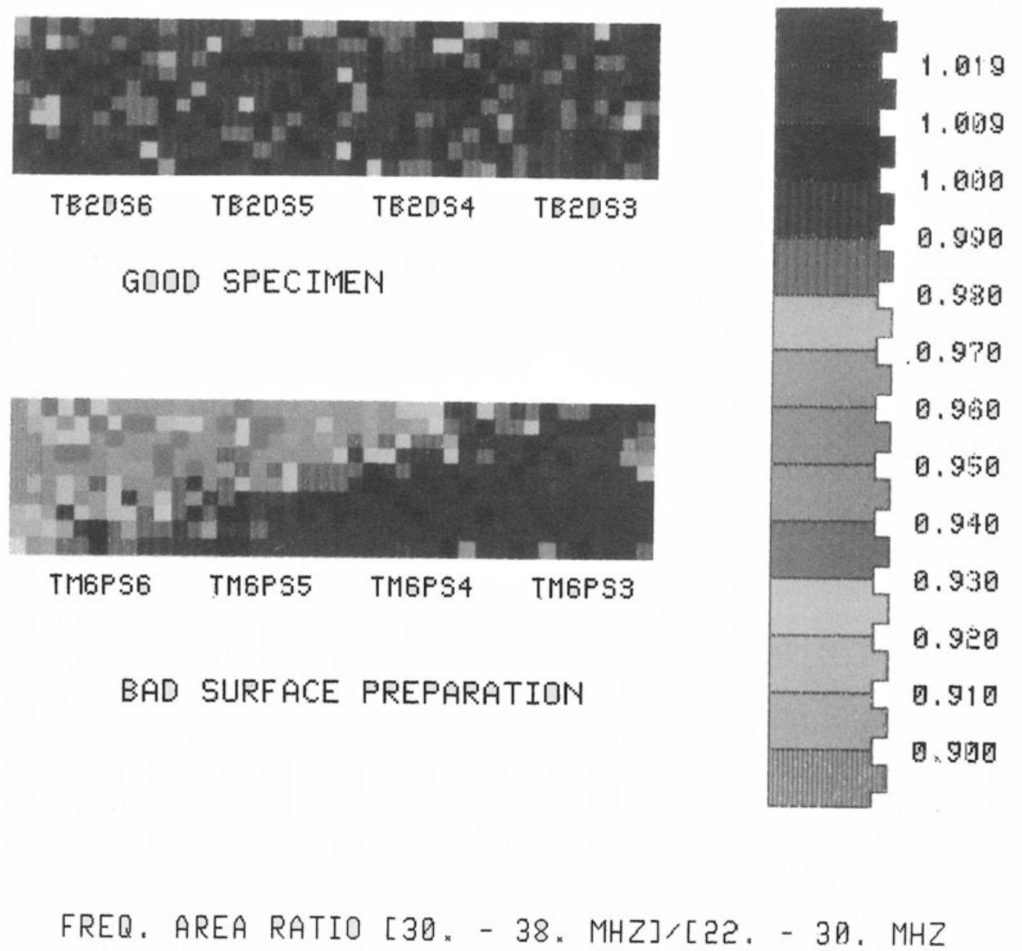

Figure 5: Ultrasonic Feature Scans for the Detection of Good and Bad Surface Preparation.

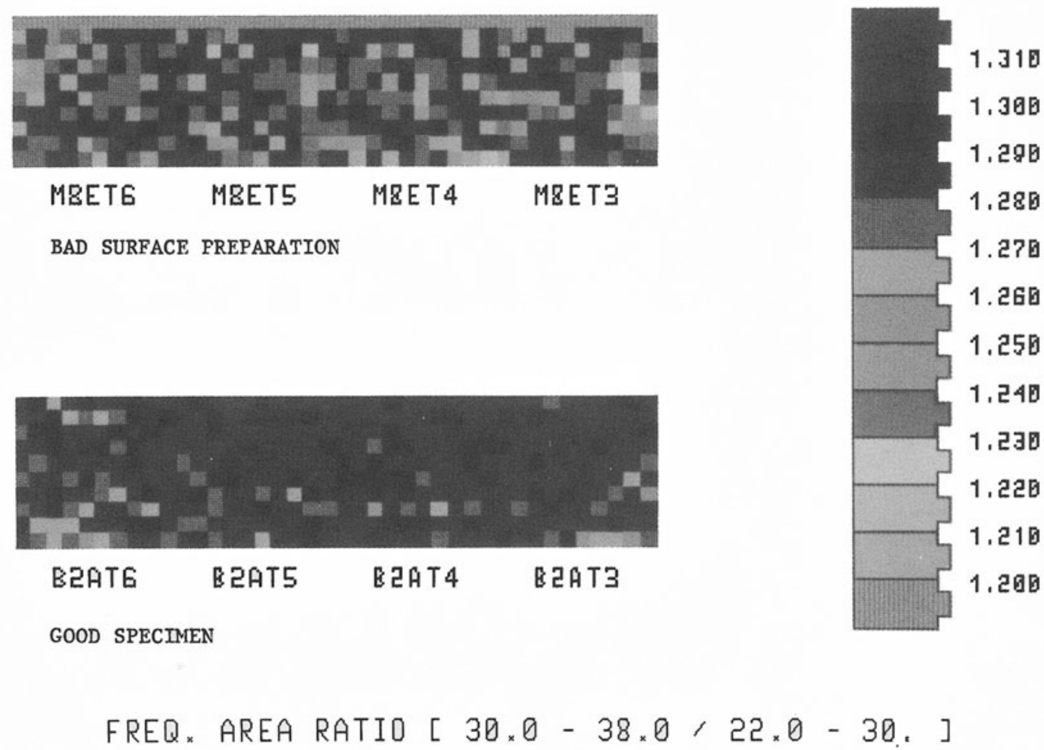

Figure 6: Ultrasonic Feature Scans for the Confirmation of Results Found in Figure 5.

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